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# Transport properties of magnetic field/liquid assisted textured $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ thick films

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Textured  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  thick films (thickness  $\approx 1$  mm) were fabricated by vacuum filtration in an applied magnetic field ( $H=7$  T). Platinum (1 wt %) was incorporated to induce liquid phase-assisted densification in films fired between 960 and 1030 °C in oxygen. The transport critical current densities ( $J_{ct}$ ) of films fired to 1030 °C exhibited nearly field insensitive behavior between  $H=0-3$  T ( $H\parallel c$ -axis) at 77 K, with  $J_{ct}\approx 2500$  A/cm<sup>2</sup> and corresponding critical currents ( $I_c$ ) of nearly 100 A at  $H=1$  T. © 1995 American Institute of Physics.

The discovery of the superconductor  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  (Y123) (Ref. 1) with its transition temperature ( $T_c$ ) above the boiling point of liquid nitrogen has generated much interest in the development of high  $T_c$  components for applications at 77 K. The attractive intrinsic properties of Y123 at  $T\leq 77$  K, include its low thermal flux creep and high upper critical field in excess of 500 T. Several techniques have been developed to produce textured  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  (Y123) bulk,<sup>2</sup> single domain,<sup>3-6</sup> and thin film<sup>7,8</sup> components with superior superconducting properties. Magnetic alignment was demonstrated early on as an effective means of texturing Y123.<sup>9</sup> This approach, however, was largely abandoned because samples exhibited weak-link behavior resulting from conventional, solid-state densification.<sup>2</sup> Melt texturing via melt-growth<sup>3-5</sup> or directional solidification<sup>10,11</sup> processes has also gained attention; however, both techniques have inherent limitations. Melt-growth processing yields large, textured Y123 single domains which are size limited (maximum diameter  $\approx 100$  mm) or multidomain samples that are poorly connected and misaligned. Alternatively, directional solidification of Y123 produces highly oriented samples, but at slow growth rates ( $\approx 1$   $\mu\text{m/s}$ ). Recently, textured Y123 thin films have been fabricated on biaxially aligned oxide buffer layers deposited by ion beam assisted deposition (IBAD) on flexible metal alloys.<sup>7,8</sup> Such films exhibit excellent crystallographic orientation and high transport current densities (e.g.,  $J_{ct}>10^5$  A/cm<sup>2</sup> at 77 K). Furthermore, this approach appears amenable to scale-up, thus renewing interest in the use of Y123 superconductors for large scale applications at 77 K.

We have demonstrated that magnetic alignment of Y123 particles under ambient conditions followed by liquid phase-assisted densification in the presence of platinum (Pt) yields dense, textured Y123 thick films.<sup>12,13</sup> Hereinafter the technique is referred to as the magnetic field/liquid assisted textured (MFLAT) process. In this letter, we report on the current carrying properties of MFLAT processed  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  thick films which exhibit magnetic field insensitive behavior at elevated temperatures in modest applied fields ( $T=77$  K,  $H=0-3$  T).

Y123 thick films were formed by vacuum filtration of a particulate suspension in an applied magnetic field ( $H=7$  T)

as shown in Fig. 1. 10 vol %  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  particles (diameter  $\approx 2-6$   $\mu\text{m}$ ) were dispersed by dissolving an appropriate amount of an organic dispersant in isopropyl alcohol. Cast films (diameter=2.5 cm, thickness  $\approx 1.0$  mm) contained 1 wt % platinum (Pt) and were fired on magnesium oxide (MgO) single crystal substrates with (100) orientation. Liquid-assisted densification was carried out by heating samples in nitrogen to 600 °C at 1 °C/min, holding at 600 °C for 2 h in oxygen, heating to 950 °C at 5 °C/min, holding at 950 °C for 2 h, heating to a maximum temperature of 1010 or 1030 °C at 5 °C/min in oxygen, rapidly cooling to 1010 °C, and then slow cooling to 950 °C at 1 °C/h. Representative samples underwent solid-state densification following an analogous thermal profile to 950 °C with a 24 h hold at temperature. All samples were furnace cooled from 950 to 450 °C, oxygen annealed for 24 h, and cooled to room temperature.

We have shown that Pt has a dramatic impact on the melting behavior of Y123, reducing the onset of incongruent melting by  $\approx 70$  °C in oxygen.<sup>12,13</sup> For Y123 (+1 wt % Pt) films, liquid phase-assisted densification occurs between 960–1030 °C in pure oxygen. In this temperature range,

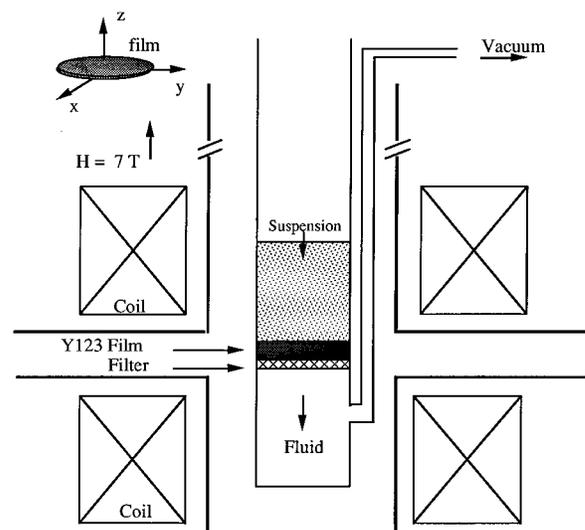


FIG. 1. Schematic cross-sectional view of magnetic field-assisted processing technique. (Note: Split coil superconducting magnet has vertical and radial warm bores.)

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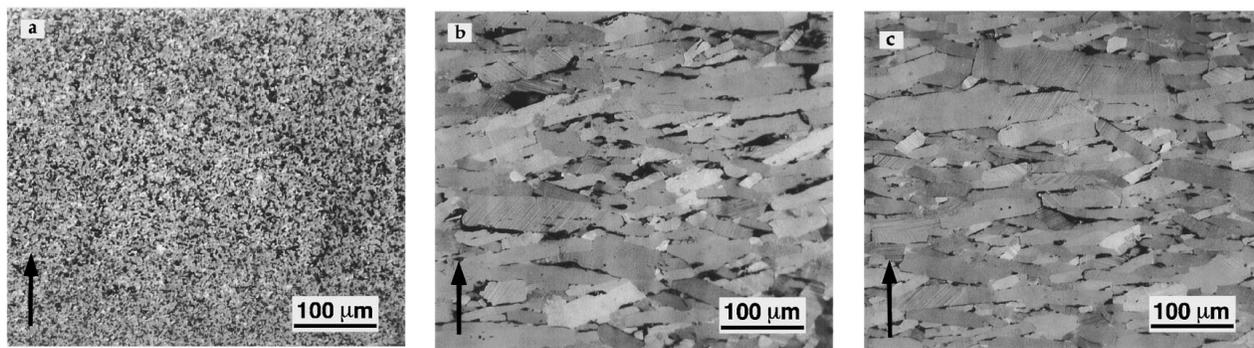


FIG. 2. Optical micrographs of polished cross sections of textured Y123 films fired on MgO substrates heat treated at maximum temperatures of (a) 950 °C, (b) 1010 °C, and (c) 1030 °C. (Arrow denotes  $c$ -axis direction of each film.)

Y123 is partially decomposed to form a liquid phase and nonsuperconducting phases (e.g.,  $Y_2BaCuO_5$ ). Unmelted, prealigned Y123 particles serve as seeds for textured growth during densification. In comparison, melt processing requires processing temperatures above 1030 °C (in  $O_2$ ) and fully decomposes the Y123 phase.

Figures 2(a)–2(c) show optical micrographs of polished cross sections of textured Y123 thick films fired on MgO to  $T_{max}=950, 1010,$  and  $1030$  °C, respectively. Films were removed from the substrate prior to microstructural analysis. Y123 thick films heated above 960 °C have an average grain size  $>50$   $\mu m$  (long dimension) and are nearly 100% dense, features indicative of a liquid-assisted densification process. In contrast, the conventional solid-state sintered ( $T_{max}=950$  °C) Y123 films are porous, fine grained samples.

Hysteresis measurements of aligned Y123 films were made using a high-field SQUID magnetometer (model MPMS, Quantum Design, San Diego, CA) at 5 K and were normalized with respect to sample volume. Representative  $M$ – $H$  loops are shown in Fig. 3 for samples fired to  $T_{max}=1010$  °C. The bulk anisotropy (or degree of  $c$ -axis texture) within these films was evaluated from the ratio of  $\Delta M$  ( $H \parallel c$ -axis) to  $\Delta M$  ( $H \perp c$ -axis). This ratio ( $\Delta M_{\parallel} / \Delta M_{\perp}$ ) was averaged over the applied magnetic field range from 1 to 5 T for each film and was found to be  $\approx 2.9$  ( $T_{max}=1030$  °C) to 1.5 ( $T_{max}=950$  °C). These values lie between those measured for randomly oriented ( $R=1$ ) and single crystal ( $R \approx 10$ ) Y123 samples.<sup>14</sup> Notably, the presence of the liquid phase during densification leads to a twofold increase in the bulk  $c$ -axis texture of the Y123 thick films relative to those conventionally sintered. Since the bulk texture of as-vacuum filtrated samples is similar to the conventionally sintered ones, it is believed that further improvements in final grain alignment are possible upon optimization of the casting procedure.

Transport measurements were carried out using a continuous dc four-point probe technique with an electric field criterion of 1  $\mu V/cm$ . A schematic view of the sample geometry (approximate dimensions are given) is shown in the inset of Fig. 4. Silver contacts were made on transport samples cut from densified thick films. Samples were annealed at 450 °C for 24 h in oxygen prior to the measurements. The contact resistivity was about 10  $\mu\Omega$  cm at 77 K. Transport samples were mounted on the testing fixture of a variable

temperature insert (VTI) apparatus ( $T=77$  K), which was placed in the vertical bore of the split-coil 7 T superconducting magnet shown in Fig. 1. The magnetic field was ramped to a maximum value of  $H=1$  or 5 T, and then reduced by increments of 0.2 or 1 T, respectively. Transport measurements were performed on several samples for each set of conditions and average values are reported. Due to problems arising from contact failure, individual samples were generally not evaluated over the entire magnetic field range.

The dependence of transport critical current density ( $J_{ct}$ ) on applied magnetic field is shown in Fig. 4 for films fired to different maximum temperatures. Y123 thick films fired to 950 °C exhibited the characteristic weak-link behavior associated with conventionally sintered, polycrystalline samples. In contrast, magnetic field insensitive transport properties were observed for films fired to  $T_{max}=1010$  and 1030 °C between  $H=0$ –1 T and  $H=0$ –3 T at 77 K, respectively. The average  $J_{ct}$ 's of samples fired to 1030 °C ranged from 2100 to 3000 A/cm<sup>2</sup> between 0–3 T at 77 K, with corresponding critical currents ( $I_c$ ) of nearly 100 A at 1 T.

To our knowledge, the MFLAT process is the first technique to yield bulk Y123 samples which exhibit field insensitive properties at 77 K in modest applied magnetic fields ( $H=0$ –3 T). The transport properties of these Y123 thick

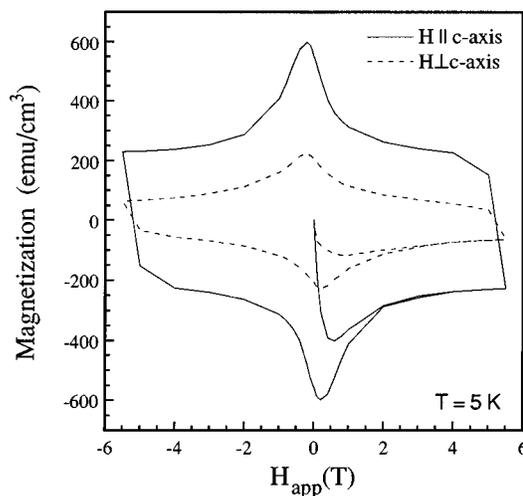


FIG. 3. Magnetization hysteresis loops at 5 K of Y123 thick films heated to  $T_{max}=1010$  °C.

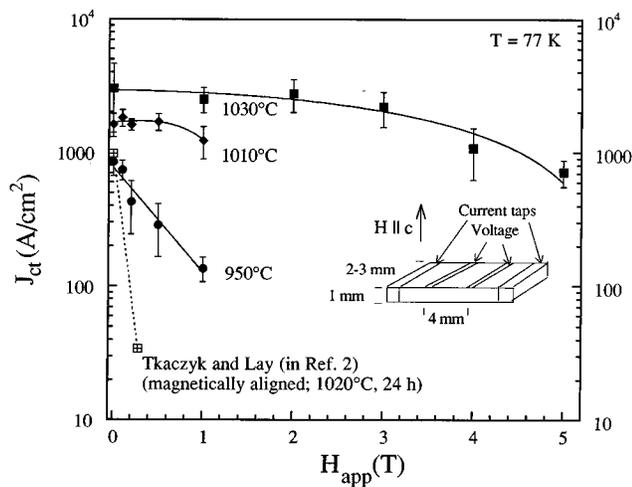


FIG. 4. Transport critical current density ( $J_{ct}$ ) as a function of applied magnetic field ( $H||c$ -axis) for Y123 thick films heated to different maximum temperatures.

films compare favorably to those obtained for samples fabricated by other techniques. For example, the transport properties of magnetically-aligned bulk Y123 samples conventionally sintered at 1020 °C (no Pt added) by Tkaczyk and Lay<sup>2</sup> are also shown in Fig. 4. Their properties clearly display a strong magnetic field dependence. Since they have similar bulk texture, we attribute the observed differences mainly to differences in sample density, grain size, and connectivity.<sup>3</sup> The MFLAT process produced dense, large-grained samples (refer to Fig. 2); whereas those prepared by Tkaczyk and Lay had both lower density ( $\rho \leq 88\%$  theoretical density) and average grain size ( $\approx 20 \mu\text{m}$ , long dimension).<sup>2</sup> Jin *et al.*<sup>3</sup> were the first to demonstrate that transport properties of Y123 samples densified in the presence of a liquid phase exhibited a lower magnetic field dependence relative to conventionally sintered samples. Their dense, bulk Y123 samples had local grain alignment (average size  $\approx 40\text{--}600 \mu\text{m}$  in length) and a maximum  $J_{ct} \approx 1000 \text{ A/cm}^2$  at 77 K, 1 T. The nearly threefold increase in the transport properties of the MFLAT Y123 films is believed to result from their global  $c$ -axis alignment.

The transport properties of MFLAT Y123 thick films are similar to those reported for Y123 thin films deposited on polycrystalline substrates, which have  $J_{ct}$  values between  $10^3$  and  $10^4 \text{ A/cm}^2$  at 77 K, 0 T,<sup>15,16</sup> but are far below those reported for biaxially aligned Y123 films, where  $J_{ct} > 10^5 \text{ A/cm}^2$  at 77 K, 0 T.<sup>7,8</sup> Such observations can be attributed to the lack of azimuthal ordering of the in-plane  $a$  and  $b$  axes in uniaxially aligned films. Dimos *et al.*<sup>17,18</sup> have shown that when in-plane misorientation angles exceed a few degrees between neighboring Y123 grains, their boundaries have the properties of Josephson weak links. Thus, the presence of high angle grain boundaries in these MFLAT films may limit the magnitude of transport critical current density, but does not contribute to significant degradation of  $J_{ct}$  in modest applied fields at 77 K.

Critical current,  $I_c$ , is an important figure of merit for large-scale superconducting applications. Due to film thickness limitations, thin film superconductors may require biax-

ial alignment to meet the demands by large current specifications. The highest reported  $I_c$ 's for biaxially aligned Y123 films (thickness =  $2 \mu\text{m}$ ) were recently announced by Foltyn *et al.*<sup>19</sup> to be approximately 50 A at 75 K, 1 T. In comparison, MFLAT Y123 thick films which do not have processing-imposed thickness limitations, carry nearly twice this  $I_c$  value under analogous conditions. A scaled-up MFLAT process may be competitive with the biaxial thin film approach when considering both superconducting properties and economic factors. The feasibility of continuous, in-field processing utilizing the radial warm bore of the split coil superconducting magnet to produce doctor-blade MFLAT Y123 tapes is under investigation.

In summary, we have demonstrated that textured  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  thick films with good current carrying capability can be fabricated by a magnetic field/liquid-assisted texturing process. Such films exhibited nearly magnetic field insensitive behavior between  $H=0\text{--}3 \text{ T}$  ( $H||c$ -axis) at 77 K, with  $J_{ct} \approx 2500 \text{ A/cm}^2$  and corresponding critical currents ( $I_c$ ) of nearly 100 A at  $H=1 \text{ T}$ . The MFLAT technique appears to be a promising new approach for producing Y123 thick films for large-scale applications in modest applied fields.

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